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Method of manufacturing antimony trioxide powder

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(54) Name of invention: Method of manufacturing antimony trioxide powder

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Specifications

1. Name of invention

Method of manufacturing antimony trioxide powder

2. Scope of patent application

The method of manufacturing antimony trioxide powder characterized by the fact that while using the method of manufacturing antimony trioxide powder whereby metallic antimony is melted by heat, whereby the antimony is oxidized and volatilized through air being blown upon the molten metal surface and whereby the gas present over the molten metal surface is sucked in and cooled, it also lowers the gas-sucking pipe according to the drop in the molten metal surface level, injects simultaneously cooling air in a volume equal to or higher than that of the air used for oxidation in a place inside the said suction pipe 0.3-0.6 m removed from the molten metal surface and so cools rapidly the sucked-in gas.

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3. Detailed explanation of the invention

(Field of industrial use)

The present invention concerns a method of manufacturing fine antimony trioxide powder.

(The conventional technique)

When using antimony trioxide for fire retardation in plastic, a powder of a grain diameter as small as possible is desirable because of the need for good dispersibility. In the past, in order to manufacture antimony trioxide powder to be used for the above purpose, metallic antimony was melted in a crucible oven, the antimony was oxidized and volatilized through air blown on the surface of the molten metal, the gas present over the molten metal surface was sucked up and driven to a collector, and the oxide powder deposited through natural cooling was then captured. However, when such method was used, the average particle size of the obtained antimony trioxide was always large, and the particle size distribution was invariably wide. Therefore several collectors were disposed in series so that coarser dust was collected near the oven and so that the dust particles grew smaller as the distance from the oven increased. With this system, the powder recovered in the collector at the final stage is only abour 0.7 μ in diameter. Moreover, the fine powder obtained at the final stage represents less than 20% of the total.

(The problems which the present invention aims to solve)

The present invention was made with the above facts in mind. It proposes a method making it possible to manufacture at a high yield fine antimony trioxide.

(The means of solving the problems)

In order to reach this goal the present inventors wondered whether it would not be possible to control the solidification of the antimony 112
trioxide vapour through forced cooling of the sucked-in gas, and they performed tests introducing cooling air at various places of the gas suction pipe. They found that a good yield of fine antimony powder could be obtained by introducing a given minimum volume of air at a place remote by a determined distance from the molten metal surface, and so they came up with the present invention. This means that the present invention is characterized by the fact that the gas suction pipe containing the antimony trioxide vapour is lowered according to the drop of the molten metal surface level, that cooling air of a volume equal or superior to that of the oxidizing gas is injected inside the said suction pipe at a place 0.3-0.6 m removed from the molten metal surface and by the fact that the sucked-in gas is rapidly cooled.

(Operation)

The reason why the place where the cooling air is being injected inside the gas suction pipe is from 0.3 to 0.6 m removed from the molten metal level, is that the particle size of the obtained antimony trioxide powder increases when such place becomes too remote from the molten metal surface, and that 0.6 m is the limit for the obtention of a 1 μ particle size. If the volume of cooling air is the same, the particle size will decrease as the injection site grows nearer to the molten metal surface.

However, if the injection site is too close to the molten metal surface, then cooling air will leak from the suction opening of the gas suction pipe into the furnace. This risks to trigger undesirable temperature variations inside the furnace so that the limit is about 0.3 m.

The reason why the cooling air injected into the gas suction pipe must be of a volume equal or superior to that of the oxidizing air is that the grain size cannot reach 1 μ at the lowest 0.3 m limit for the injection site if the volume of cooling air is lower. The particle size of the antimony trioxide powder obtained grows smaller as the volume of cooling air increases. So, by aptly combining this volume of cooling air with the above cooling air injection site, it becomes possible to obtain antimony trioxide powder with optional particle sizes of or below 1 μ.

As the molten metal surface level of the molten antimony trioxide drops gradually because of the oxidizing volatilization, the air injection port must be gradually lowered according to the drop in molten surface level so as to ensure a constant distance from the molten metal surface. If in this case the injection port in question becomes lower than the suction opening of the gas suction pipe, cooling air will be admixed into the furnace, so it is necessary to lower also the gas suction pipe in question in keeping with the drop in the molten metal surface level. To achieve such lowering, one may adopt a structure whereby the gas suction pipe and the cooling air injection port are suspended from the upper part of the furnace.

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It goes without saying that in order to prevent dispersion of the powder, the gas suction volume of the gas suction pipe must be a little greater than the sum of the oxidizing air and of the cooling air volumes.

(Practical example)

We loaded 800 kg of metallic antimony in a crucible oven with a 0.1 m^3 internal capacity, and melted by heating at 1,200°C. We directed 1 Nm^3/min of oxidizing air to the molten metal surface and sucked up the gas in the furnace (temperature: 1,020°C) into a gas suction pipe of a 15 cm diameter inserted through the lid of the furnace. We injected cooling air (temperature: 20°C) at the rate of 1-5 Nm^3/min from a sprayer 5 cm in diameter and 8 cm in height provided with numerous small perforations on its sides. The sprayer had been fitted inside the suction pipe in question with its lower end positioned 0.3 m over the surface of the molten metal. The volume of sucked-in gas was set at 4-8 Nm^3/min, and the produced antimony trioxide powder was trapped with a pack filter. The volume trapped was about 40 kg per every hour. We measured the average particle size of the antimony trioxide powder obtained with a specific surface area gauge. Table 1 shows the volumes of cooling air and the average grain size measurement results.

Table 1 shows that the volume of cooling air must be at least equal to the volume of the oxidizing air in order to obtain a grain size of 1 μ or lower. We see that the average grain size of the obtained antimony trioxide decreases as the cooling air becomes more abundant.

(Effectivity of the invention)

By the method of the present invention, we were able to establish a technique yielding a high rate of antimony trioxide powder having an optional particle size with a maximum average of 1 μ.

Patent applicant: Sumitomo Metal Mining Co., Ltd.

a	b	c
実験番号	冷却用空気（Nm³/分）	Sb_2O_3 平均粒径(μ)
1	1	1.0
2	2	0.55
3	3	0.4
4	4	0.2
5	5	0.1

Table 1

a. test number

b. cooling air (Nm^3/min)

c. Sb_2O_3 average grain size (μ)